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IS 7588 (1992): Barytes for explosives and pyrotechnic composition [CHD 26: Explosives and Pyrotechnics]



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भारतीय मानक

विस्फोटक एवं आतिशबाजी संघटनों के लिए
बैराइट्स — विशिष्ट

(पहला पुनरीक्षण)

Indian Standard

BARYTES FOR EXPLOSIVES AND
PYROTECHNIC COMPOSITIONS —
SPECIFICATION

(First Revision)

UDC 622.368.92 : 662.1/.4

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

Barytes, a naturally occurring form of barium sulphate, is widely used as an inert ingredient in the explosive and pyrotechnic formulations. For a long time now the explosive manufacturers and formulators have been experiencing difficulty in procuring suitable quality of raw materials. Indian Standards already published on this subject cater to the requirements of rubber and chemical industries and oil-well drilling. This standard has been formulated with a view to facilitate procurement of assured quality of barytes for explosive and pyrotechnics industry.

This standard follows in the series of Indian Standards on barytes. Other standards on this subject are:

IS 1683 : 1960 Barytes for rubber industry, and

IS 2881 : 1964 Barytes for chemical industry and oil-well drilling.

This standard was originally published in 1974. On the basis of the experience gained since this standard was first published, it has been revised to widen its scope to include two additional grades of barytes for use by the Defence, namely, barytes — ammunition grade and barytes — for use as an ingredient of high explosive (HE) substitutes.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

BARYTES FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for barytes intended for use in explosive and pyrotechnics compositions.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	Title
75 : 1973	Linseed oil, raw and refined (<i>second revision</i>)
265 : 1987	Hydrochloric acid (<i>third revision</i>)
323 : 1959	Rectified spirit (<i>revised</i>)
878 : 1975	Measuring cylinders (<i>first revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
1683 : 1973	Barytes for rubber industry (<i>first revision</i>)

3 GRADES

The material shall be of the following grades:

- Grade 1 — Barytes, for general use in explosive and pyrotechnic industry
- Grade 2 — Barytes—Ammunition grade, for use by Defence
- Grade 3 — Barytes, for use as an ingredient of high explosive (HE) substitutes by Defence.

4 REQUIREMENTS

4.1 Description

The material shall consist essentially of barium sulphate, bleached or unbleached, in dry powder form. It shall be white to off-white in colour and free from all extraneous matter.

4.2 The material of all the three grades shall comply with the specific requirements for individual grades laid down in Table 1 when tested according to the test methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of Table 1.

5 PACKING AND MARKING

5.1 Packing

Unless agreed to otherwise between the purchaser and the supplier, the material shall be packed in clean jute bags with a polyethylene liner.

5.2 Marking

The packages shall be marked legibly and indelibly with the following information:

- a) Name and description of the material,
- b) Net mass of the material,
- c) Indication of the source of manufacture, and
- d) Lot number in code or otherwise to enable the batch of manufacture to be traced back from records.

5.2.1 Each package may also be marked with the 'Standard Mark'.

6 SAMPLING

6.1 Preparation of Test Samples

Representative samples of the material shall be prepared as prescribed in Annex A of IS 1683 : 1973.

6.2 Number of Tests

6.2.1 Tests for the determination of pH, grit contents, fineness and bulk density shall be conducted on each of the individual sample.

6.2.2 Tests for the determination of all the remaining characteristics given in Table 1 shall be carried out on the composite sample.

6.3 Criteria for Conformity

6.3.1 For Individual Samples

For declaring the conformity of the lot:

- $X + 0.6 R$ shall be less than or equal to the maximum specified requirements, and
- $X - 0.6 R$ shall be greater than or equal to the minimum specified requirements.

where

X = mean value of test results, and

R = range of results.

6.3.2 For Composite Sample

For declaring the conformity of the lot to the

requirements of all the characteristics tested on the composite sample, the test results shall satisfy the corresponding specified requirements.

Table 1 Requirements for Barytes for Use in Explosives and Pyrotechnic Compositions
(Clauses 4.2 and 6.2.2)

Sl No.	Characteristic	Requirement for			Method of Test (Ref to Cl No. in Annex A)
		Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Barium sulphate, percent by mass, <i>Min</i>	90.0	96.0	96.0	A-2
ii)	Matter soluble in dilute hydrochloric acid, percent by mass, <i>Max</i>	3.0	—	1.0	A-3
iii)	Matter soluble in water, percent by mass, <i>Max</i>	0.5	0.5	0.1	A-4
iv)	pH (of aqueous extract)	6 to 9	7	7	A-5
v)	Carbonates (as CaCO_3), percent by mass, <i>Max</i>	1.0	0.5	0.5	A-6
vi)	Grit, percent by mass, <i>Max</i>	0.001	Nil	Nil	A-7
vii)	Loss on drying (at 105 to 110°C), percent by mass, <i>Max</i>	0.5	0.5	0.5	A-8
viii)	Bulk density, g/ml	2.5 (<i>Min</i>)	—	4.0 to 4.5	A-9
ix)	Fineness (wet sieving) :				A-10
	Material retained on 125-micron IS sieve, percent by mass, <i>Max</i>	0.002	0.25	—	
	Material retained on 63-micron IS sieve, percent by mass, <i>Max</i>	1.0	—	Nil	
x)	Oil absorption, percent by mass	—	6 to 12	—	A-11
xi)	Acidity (as H_2SO_4), percent by mass, <i>Max</i>				A-12
	a) to methyl orange	—	—	Nil	
	b) to phenolphthalein	—	—	0.05	
xii)	Alkalinity (as Na_2CO_3), percent by mass, <i>Max</i>				A-12
	a) to methyl orange	—	—	0.05	
	b) to phenolphthalein	—	—	Nil	

ANNEX A

(Clause 4.2)

METHOD OF TEST FOR BARYTES FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 PRINCIPLE

The material is fused with sodium carbonates, the fused mass is leached out with water to

remove soluble sodium compounds including silicates and filtered off. Barium remains insoluble as carbonate together with the heavy metals. The residue is treated with sodium carbonate solution to remove the adhering sulphates. The insoluble carbonates are treated with dilute hydrochloric acid till the precipitate completely dissolves. Barium is precipitated from the acidic solution as barium sulphate with the addition of ammonium sulphate which is ignited and weighed.

A-2.1 Reagents

A-2.1.1 *Sodium Carbonate* — anhydrous.

A-2.1.2 *Sodium Carbonate Solution* — 2 g/l.

A-2.1.3 *Dilute Hydrochloric Acid* — 1 : 4.

A-2.1.4 *Ammonium Hydroxide* — 20 percent, (m/m).

A-2.1.5 *Concentrated Hydrochloric Acid* — See IS 265 : 1987.

A-2.1.6 *Methyl Orange Indicator Solution*

Dissolve 0.1 g of methyl orange in 100 ml of water.

A-2.1.7 *Ammonium Sulphate Solution* — 3 percent.

A-2.2 Procedure

A-2.2.1 Weigh about 8 g of sodium carbonate. Place an appropriate quantity of it in a platinum crucible so as to form a layer about 6 mm thick. Separately, weigh accurately about 1 g of the finely powdered material and place it on top of the sodium carbonate layer in the crucible. Mix both with a glass rod. Add the rest of sodium carbonate also to the crucible. Cover and fuse the mixture over a Meker burner for 40 minutes. The fusion is started with a low flame which is gradually raised to full blast. This precaution is necessary to prevent loss due to overflowing.

Cool and as the melt cools, rotate the crucible so that the fused mass solidifies in a thin layer. This will shorten the time required for leaching. Leach out the fusion with 200 ml of hot water in a 400-ml beaker. Filter through a 15-cm filter paper (Whatman No. 40 or equivalent). Wash several times by decantation. Remove the crucible from the beaker, transfer the insoluble carbonates to the filter, wash with hot sodium carbonate solution and test after the twelfth washing to be certain that sulphates have been removed completely.

A-2.2.2 Cover the funnel containing the insoluble carbonates with a watch-glass and add hot dilute hydrochloric acid carefully, in small portions at a time to prevent loss, catching the solution in a 600-ml beaker. Add hot dilute hydrochloric acid to the platinum crucible and the beaker in which the leach was made and pour over the filter. Wash the paper with hot water until free from chlorides.

A-2.2.3 Neutralize this solution with ammonium hydroxide using methyl orange as indicator. Add 0.4 to 0.6 ml of concentrated hydrochloric acid. Dilute to 400 ml with hot water, bring the solution to boiling and add 25 ml of hot ammonium sulphate solution dropwise with constant stirring to prevent co-precipitation of calcium and magnesium. Transfer the beaker to a warm hot plate and allow to stand for at least 4 hours.

Filter on an ignited and weighed Gooch crucible, wash with hot water several times by decantation. The beaker should be scrubbed thoroughly to remove any adhering precipitate. Continue the washing until they are free from chlorides. Ignite the crucible in a muffle furnace for 35 minutes at 850°C. Cool in a desiccator and weigh. Make a blank determination in a similar manner with an equal amount of sodium carbonate and other reagents.

A-2.3 Calculation

$$\text{Barium sulphate, percent by mass} = 100 \frac{(A - B)}{M}$$

where

A = mass, in g, of the precipitate with the material,

B = mass, in g, of the precipitate in the blank determination, and

M = mass, in g, of the material taken for the test.

A-3 DETERMINATION OF MATTER SOLUBLE IN DILUTE HYDROCHLORIC ACID

A-3.1 Reagents

A-3.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

A-3.2 Procedure

Weigh accurately about 2 g of the material into a 50-ml beaker, add 50 ml of dilute hydrochloric acid. Boil for 10 minutes, dilute to 100 ml and allow to stand overnight and then filter through a filter paper (Whatman No. 42 or equivalent). Wash the filter paper with hot water until the filtrate is free from chlorides. Dry the filter paper and transfer it into a weighed crucible previously ignited. Heat the crucible on a burner and subsequently in a muffle furnace at 850°C. Weigh the crucible and the contents after cooling in a desiccator and repeat this process of heating, cooling and weighing until constant mass.

A-3.3 Calculation

$$\text{Matter soluble in dilute hydrochloric acid, percent by mass} = 100 \times \frac{M - M_1}{M}$$

where

M = mass, in g, of the material taken for the test; and

M_1 = mass, in g, of the ignited residue.

A-4 DETERMINATION OF MATTER SOLUBLE IN WATER

A-4.1 Procedure

Weigh accurately about 100 g of the powdered material. Wet it thoroughly with a small

quantity of water and then add 200 ml of freshly boiled and cooled water. Boil for 5 minutes. Cool the mixture to room temperature. Make up to 250 ml with freshly boiled and cooled water, shake and filter. Reject the first 50 ml portion of the filtrate. Take a 100 ml portion from the rest of the filtrate in a tared platinum dish and evaporate to dryness on a water-bath. Dry the residue obtained to constant mass in an oven at 105 to 110°C.

A-4.2 Calculation

$$\text{Matter soluble in water, percent by mass} = \frac{250 A}{M}$$

where

A = mass, in g, of the residue; and

M = mass, in g, of the material taken for the test.

A-5 DETERMINATION OF pH

A-5.1 Weigh 2.0 g of the material and transfer it completely to a 250-ml beaker. Add 100 ml of carbon dioxide-free water and stir for 30 minutes. Allow the solid material to settle and then filter. Collect the filtrate and determine its pH by any suitable method. Use of glass electrode system is preferable.

A-5.1.1 In case of dispute, the method using glass electrode system shall be adopted.

A-6 DETERMINATION OF CARBONATES

A-6.0 Outline of the Method

The material is heated with sufficient water and the carbonates in aqueous suspension are reacted with a known excess of standard hydrochloric acid. The excess acid is then back titrated with standard sodium hydroxide solution and the carbonate is calculated as calcium carbonate.

A-6.1 Reagents

A-6.1.1 *Standard Hydrochloric Acid* — 0.1 N.

A-6.1.2 *Standard Sodium Hydroxide Solution* — 0.1 N.

A-6.1.3 *Bromophenol Blue Indicator Solution* — 0.1 g in 100 ml of rectified spirit.

A-6.2 Procedure

Weigh accurately about 10 g of the material in a 500-ml conical flask and add about 200 ml of previously boiled water. Run in from burette a known excess of standard hydrochloric acid. Titrate the excess acid with standard sodium hydroxide solution using bromophenol blue as indicator. Carry out side by side a blank determination following exactly the same procedure.

A-6.3 Calculation

$$\text{Carbonate (as CaCO}_3\text{), percent by mass} = \frac{(A-B) \times N \times 50 \times 100}{1\,000 \times M}$$

where

A = volume, in ml, of standard sodium hydroxide solution required for blank;

B = volume, in ml, of standard sodium hydroxide solution required for sample;

N = normality of standard sodium hydroxide solution; and

M = mass, in g, of the material taken for the test.

A-7 DETERMINATION OF GRIT

A-7.0 Outline of the Method

The portion retained on 75 micron IS sieve is boiled with aqua regia. The insoluble residue obtained after digestion with aqua regia is dried. The dried residue is then sieved through 125 micron IS sieve and the portion retained on the sieve is weighed. If the residue (retained on 125 micron IS sieve) scratches soda glass then the mass previously obtained is expressed as grit.

A-7.1 Apparatus

A-7.1.1 *Sieves* — 75 micron and 125 micron IS sieves.

A-7.1.2 *Glass Slides* — soda glass.

A-7.2 Reagents

A-7.2.1 Aqua Regia

Prepared by mixing 3 volumes of concentrated hydrochloric acid with one volume of concentrated nitric acid.

A-7.3 Sieve 100 g of the material through 75 micron IS sieve with 25 mm brush. Boil the portion retained on the sieve with about 50 ml of aqua regia in a 150-ml beaker (covered with a watch-glass). Cool the beaker, dilute the acid with water and then decant the diluted acid carefully. Wash the residue remaining at the bottom of the beaker three times with water and dry the residue in an air-oven.

A-7.3.1 Sieve the dried residue carefully through 125-micron IS sieve with a light camel hair brush. Weigh out accurately the portion retained on the sieve and carry out the soda glass scratching test as prescribed in A-7.5. If the residue scratches soda glass, express the mass (previously obtained) as percentage grit.

NOTE — Care should always be taken not to lose any material at any stage of the test.

A-7.4 Calculation

$$\text{Grit, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M = mass, in g, of the residue scratching soda glass; and

M_1 = mass, in g, of the material taken for test.

A-7.5 Soda Glass Scratch Test

Take a portion of the residue (retained on 125-micron IS sieve) in between the two clean glass slides and press the slides by hand along the length and breadth of the glass. Examine whether any scratch on the glass is obtained. If there is any scratch on the glass then the residue is termed as grit.

A-8 DETERMINATION OF LOSS ON DRYING

A-8.1 Procedure

Weigh accurately about 10 g of the material and heat in a squat weighing bottle at 105 to 110°C. Cool and weigh till a constant mass is obtained.

A-8.2 Calculation

$$\text{Loss on drying, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = loss of mass, in g, on drying; and

M = mass, in g, of the material taken for the test.

A-9 DETERMINATION OF BULK DENSITY

A-9.1 Apparatus

A-9.1.1 Assemble the apparatus as shown in Fig. 1. The measuring cylinder shall be of 250-ml capacity (see IS 878 : 1975). The base of the measuring cylinder shall be ground flat. The distance between flat-ground part of the base of the measuring cylinder *A* and the rubber base pad *B*, when the cylinder *A* is raised to the full height, shall be 25 ± 2 mm.

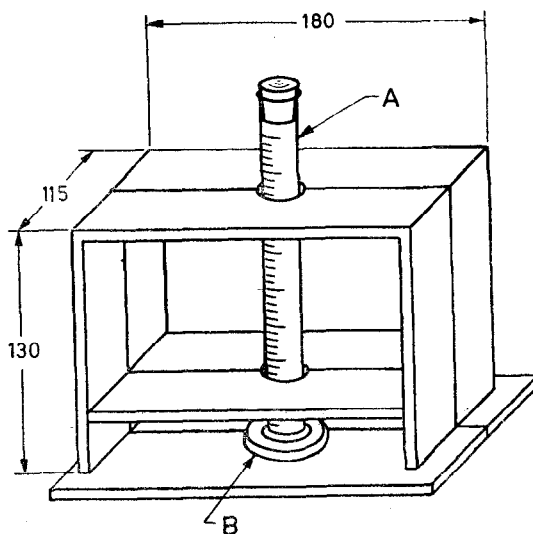
A-9.1.1.1 The rubber base pad *B* shall have a shore hardness of 42 to 50.

A-9.1.2 Pan of the balance shall be at least 10 cm in diameter and the balance shall have an accuracy of not less than 0.1 g.

A-9.2 Procedure

Place about 100 g of the material, accurately weighed, in the graduated cylinder fitted in the apparatus and tap it (at the rate of 180 drops

per minute) until a constant volume of the material is obtained. Note down the volume of the material.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR THE DETERMINATION OF BULK DENSITY

A-9.3 Calculation

$$\text{Bulk density, g/ml} = \frac{M}{V}$$

where

M = mass, in g, of the material taken for the test; and

V = final volume, in ml, of the material filling the cylinder.

A-10 DETERMINATION OF FINENESS

A-10.1 Procedure

Place about 25 g of the material, accurately weighed, on to 125 micron and 63 micron IS sieve placed one above the other, the former being on the top. Shake mechanically or by hand for 15 minutes. Wash the material on each sieve with water, gently brushing with a soft camel-hair brush until the washings are clear. Dry the residue to constant mass at $100 \pm 2^\circ\text{C}$.

A-10.2 Calculation

$$\begin{aligned} &\text{Fineness (material retained} \\ &\quad \text{on respective IS sieve),} \\ &\quad \text{percent by mass} \end{aligned} = 4M$$

where

M = mass, in g, of the residue on the sieve.

A-11 DETERMINATION OF OIL ABSORPTION**A-11.1 Apparatus****A-11.1.1 Glass Plate** — ground.**A-11.1.2 Palette Knife** — plastic.**A-11.2 Reagent****A-11.2.1 Linseed Oil** — See IS 75 : 1973.**A-11.3 Procedure**

Take about 20 g of barytes in a ground glass plate and weigh accurately (M_1). Gradually add linseed oil to it and rub the oil thoroughly onto the whole of the mass with a palette knife for about 20 minutes to obtain a coherent mass and weight it (M_2). Determine the mass of the oil used by taking the difference ($M_2 - M_1$). Determine the mass of the oil required for 100 g of dry barytes and from it, calculate the percent oil absorption.

A-12 DETERMINATION OF ACIDITY OR ALKALINITY**A-12.1 Reagents****A-12.1.1 Rectified Spirit** — See IS 323 : 1959.**A-12.1.2 Phenolphthalein Indicator Solution** — Dissolve 0.10 g in 100 ml of 60 percent rectified spirit.**A-12.1.3 Methyl Orange** — Dissolve 0.01 g in 100 ml of water.**A-12.1.4 Standard Hydrochloric Acid** — 0.10 N.**A-12.2 Procedure**

Suspend 10 g of the material in 50 ml of rectified spirit and shake for half an hour. Filter the solution and make up the volume to 100 ml with rectified spirit. Take 25 ml of this solution and add 5 drops of phenolphthalein indicator, if alkalinity (red colouration) is observed, titrate with N/10 hydrochloric acid. If no alkalinity (red colouration) is observed, add 5 drops of methyl orange indicator to another aliquot of the solution. If orange red colouration is observed, titrate against N/10 sodium hydroxide.

A-12.3 CalculationPercent alkalinity (as Na_2CO_3)

$$= \frac{TV \times 0.0053 \times 4}{10} = TV \times 0.2121$$

Percent acidity (as H_2SO_4)

$$= \frac{TV \times 0.0049 \times 4}{10} = TV \times 0.1960$$

where

 TV = titre value.

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